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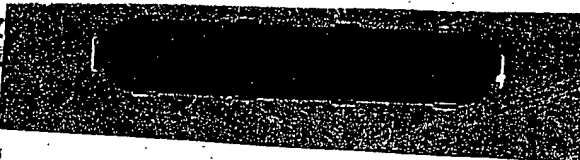
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Class	Subclass
ISSUE CLASSIFICATION	SCANNED



10-30-97  
60/064,279

SERIAL NUMBER 60/064,279 PROVISIONAL	FILING DATE 10/30/97	CLASS	SUBCLASS	GROUP ART UNIT	EXAMINER
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APPLICANTS DENNIS H. GREEN, ARVADA, CO; JEFF MUELLER, BOULDER, CO.

\*\*CONTINUING DATA\*\*\*\*\*  
VERIFIED

\*\*FOREIGN APPLICATIONS\*\*\*\*\*  
VERIFIED

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Foreign priority claimed 35 USC 119 conditions met	<input type="checkbox"/> yes <input type="checkbox"/> no <input type="checkbox"/> yes <input type="checkbox"/> no	AS FILED →	STATE OR COUNTRY CO	SHEETS DRWGS. 1	TOTAL CLAIMS	INDEP. CLAIMS	FILING FEE RECEIVED \$75.00	ATTORNEY'S DOCKET NO. 337618PROV
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Verified and Acknowledged  
Examiner's Initials  
DOUGLAS W SWARTZ  
SHERITAN ROSS  
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DENVER CO 80203

TITLE "METHOD OF REMOVING ORGANIC LIXIVIAN FROM COPPER SX EW RAFFINATE  
STREAM"

U.S. DEPT. OF COMM./PAT. & TM—PTO-436L (Rev.1)

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POSITION	ID NO.	DATE
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TYPIST	↓	2/5/78
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70110 U.S. PTO

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10/30/97

# PATENT APPLICATION



60064279

## CONTENTS

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Application \_\_\_\_\_ papers.

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549 U.S. PTO

PROVISIONAL APPLICATION COVER SHEET

a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(b)(2).

Docket Number		3376-18PROV		Type a plus sign (+) inside this box -	+
INVENTOR(s)/APPLICANT(s)					
LAST NAME GREEN MUELLER	FIRST NAME DENNIS JEFF	MIDDLE INITIAL H.	RESIDENCE (CITY AND EITHER STATE OR FOREIGN COUNTRY) 9455 WEST 68TH AVENUE, ARVADA, COLORADO 80004 3462 CRIPPLE CREEK SQUARE, BOULDER, COLORADO 80303		
TITLE OF THE INVENTION (280 characters max)					
"METHOD OF REMOVING ORGANIC LIXIVANT FROM COPPER SX-EW RAFFINATE STREAM"					
CORRESPONDENCE ADDRESS					
Douglas W. Swartz Sheridan Ross P.C. 1700 Lincoln Street, Suite 3500 Denver					
STATE	Colorado	ZIP CODE	80203	COUNTRY	United States of America
ENCLOSED APPLICATION PARTS (check all that apply)					
X	Specification	Number of Pages	17	X	Small Entity Statement
X	Drawing(s)	Number of Sheets	1		Other (specify)
METHOD OF PAYMENT (check all that apply)					
X	A check or money order is enclosed to cover the filing fees			PROVISIONAL FILING FEE AMOUNT (\$)	\$75.00
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The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

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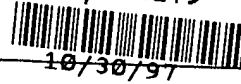
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10/30/97

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FEE RECORD SHEET

01/14/1998 JWASHINGTON 00000076 60064279  
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METHOD OF REMOVING ORGANIC LIXIVIAN  
FROM COPPER SX-EW RAFFINATE STREAM

5

FIELD OF THE INVENTION

The present invention relates generally to leaching systems and specifically to treatment systems for removing an organic lixiviant from a leaching solution.

10

THE PROBLEM

The present invention generally relates to the removal of copper ions from copper ore, and more particularly to enhanced removal of copper ions using a microfiltration or ultrafiltration process which removes entrained organic collector from the aqueous raffinate solution exiting the copper solvent extraction process. This process improves the copper hydrometallurgical mining process through more efficient copper leaching (no coating of the ore by the organic collector) and more efficient copper recovery (reduced losses of expensive organic collector).

20

The techniques used to remove copper from raw ore determine the overall efficiency of the copper mining operation. Hydrometallurgical copper mining operations using a leaching system and a copper extraction plant, such

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as a solvent extraction/electrowinning (SX/EW) plant, are now accepted processes in the copper mining industry.

Currently, electrowon copper accounts for about 30% of total U.S. copper production. Worldwide, there are more than 26

5 major heap, dump, or in-situ leaching operations using SX/EW, with a total capacity of -800,000 tons of copper annually. The industry trend continues towards this technology as higher-grade ores are depleted and smelting costs increase. Other advantages of this technology, such as the ability to process low-grade ores, low labor requirements, ease of operation in remote areas, and low operating costs, make it attractive to mining companies.

"Copper hydrometallurgy", in which copper ions are leached or otherwise extracted from raw ore using liquid chemical agents, has been of interest since as early as the 15 17<sup>th</sup> century when copper recovery methods involving iron precipitating agents from sulfuric acid based copper solutions were tested. The hydrometallurgical circuit consists of copper leaching and copper recovery.

20 First, a copper leaching agent, "lixiviant", is selected for use in leaching copper ions from copper ore.

Representative lixiviants include but are not limited to sulfuric acid ( $H_2SO_4$ ), a combination of  $H_2SO_4$  and ferric sulfate,  $Fe_2(SO_4)_3$  (primarily for sulfide containing ore materials), acidic chloride solutions (e.g. ferric chloride,  $FeCl_3$ ) or cupric chloride,  $CuCl_2$ ), nitrate solutions, ammonia, and ammonium salt compositions. Sulfuric acid is by far the most common lixiviant. The lixiviant is applied to the ore (which is stacked or piled in a large heap or dump) via a sprinkler type system and allowed to percolate downwardly into the ore. As a result, copper ions are leached from the ore and collected within the lixiviant to generate a lixiviant product that consists of a copper ion concentration (also known as "pregnant leach solution"). The lixiviant exits the bottom of the ore and is collected. Further information regarding the lixiviant leaching process is disclosed in U.S. Patent No. 5,476,591 to Green et al., which is incorporated herein by reference.

Next, a copper recovery process is used to selectively extract copper from the collected lixiviant. Representative copper recovery processes include but are not limited to solvent extraction/electrowin (SX/EW), direct

electrowinning, ion exchange - electrowin (IX/EW), and iron precipitation. Solvent extraction/electrowin is presently the most common copper recovery process. SX/EW technology was implemented in the 1960's with the development of

5 special organic extractants for copper. The SX/EW process consists of three closed solution loops. In the first loop, the acid leach solution containing valuable copper ions and a multitude of other metal ions is fed into a mixer/settler tank where it is contacted with a copper-extracting organic  
10 liquid, commonly referred to as "lix" or "organic collector." The "lix" preferentially extracts from 70 to 90% of the copper ions from the acid leaching feed solution. The second closed loop extraction step involves contacting the loaded organic collector with an electrolyte stream from  
15 the electrowinning process. The copper ions are transferred from the organic solution or "lix" to the lean electrolyte. In the third and final closed loop, the rich electrolyte flows between a cathode plate and an insoluble anode, where 70 - 90% of the copper is removed through "plating." The  
20 electrochemical cell "plates" a stainless steel electrode with copper using an applied current. The copper plated

cathode plates are then periodically removed from the process to obtain the solid, high purity copper product.

In most common copper leaching - copper recovery operation, a strong sulfuric acid solution passes downwardly through a heap or dump of low-grade copper ore and a liquid product is produced which contains remaining amounts of acid in combination with metal ions. The copper ions are then removed from the liquid product by the SX/EW copper extraction circuit to produce the high-purity cathode copper desired by many manufacturing companies.

The copper leaching - copper recovery process must be improved to overcome inherent problems such as "carry over" of organic into the stripped PLS (raffinate). This loss of organic lixiviant can represent a large economic loss. The organic lixiviant can also create an environmental problem as it "coats" or contaminates the ore heap. A further potential problem created by the "coating" of the ore to be leached is the prevention of air exchanges to the ore to provide oxygen to the bacteria; "Thio Baocillus Fero Oxidants", in the leach system.

The present invention specifically provides an improved

method for copper recovery in which the carry over of entrained organic in the raffinate is prevented, leaving a cleaner lixiviant (sulfuric acid solution) for enhanced copper ore leaching. Accordingly, the invention represents, 5 an advance in the art of copper mining technology, as described in detail herein.

#### SUMMARY OF THE INVENTION

These and other needs are met by the process of the 10 present invention which recovers a metal from a metal-containing material. The process includes the steps of:

(a) contacting a metal leaching agent with the metal-containing material to solubilize the metal in a pregnant leach solution;

15 (b) contacting the pregnant leach solution with an organic collector to transfer at least a portion of the solubilized metal to the organic collector to form a pregnant organic solution including a metal-containing organic collector and a stripped pregnant leach solution, 20 wherein the stripped pregnant leach solution contains at least a portion of the organic collector;

(c) recovering the metal from the metal-containing organic collector; and

(d) filtering the stripped pregnant leach solution with a filter to form a concentrate containing at least most  
5 of the organic collector and a permeate. The process can further include the steps of contacting the permeate with metal-containing material and recovering the organic collector from the concentrate.

By recovering the organic collector in the concentrate,  
10 the process can reduce, or eliminate, carry over of the organic collector into the leaching step and can recover the organic collector from the concentrate for reuse. Accordingly, the process provides a large, direct economic benefit and eliminates coating of the ore to be leached with  
15 the organic collector.

The metal, organic collector, and leaching agent can be a variety of materials. The metal is preferably selected from the group consisting of copper, nickel, cobalt, uranium, zinc and mixtures thereof. The organic collector  
20 is preferably selected from the group consisting of the resins sold under the tradenames "LIX 63", "LIX 64",

LIX 65", "LIX 85" and "LIX 860" as manufactured by HORNKREL  
and "PT5050", M5640", and "M56115" as manufactured by ACORGA  
LTD. and mixtures thereof. The leaching agent is preferably  
selected from the group consisting of sulfuric acid, a  
5 chloride, a nitrate, ammonia, ammonium salts, a sulfate and  
mixtures thereof.

The filter is preferably a microfilter or an  
ultrafilter. The filter preferably has a pore size ranging  
from about .003 to about .1 micron and more preferably  
10 ranging from about .01 to about .05 microns.

The filtration step preferably causes the concentrate  
to constitute less of the stripped pregnant leach solution  
than the permeate. More preferably, the concentrate  
constitutes no more than about 20% and most preferably no  
15 more than about 5% of the stripped pregnant leach solution.  
More preferably, the permeate constitutes at least about 80%  
and most preferably at least about 95% of the stripped  
pregnant leach solution.

The permeate comprises at least most of the leaching  
20 agent in the stripped pregnant leach solution. Preferably,  
the permeate comprises at least about 60% and more

preferably at least about 95% of the leaching agent in the stripped pregnant leach solution.

The concentrate comprises at least most of the organic collector in the stripped pregnant leach solution.

5 Preferably, the concentrate comprises at least about 90% and more preferably at least about 99% of the organic collector in the stripped pregnant leach solution. Further details concerning the filtration step and the filter are set forth in U.S. Patents 5,476,591; 5,310,486; and 5,116,511, each of  
10 which is incorporated fully herein by reference.

#### DETAILED DESCRIPTION

The process improvements claimed in the present invention will result from utilizing a microfiltration or  
15 ultrafiltration membrane system to process the raffinate exiting from the copper recovery plant. As described above and shown in Figure 1, a strong leaching solution 10 passes downwardly through a heap or dump 14 of low-grade copper ore and a lixiviant 18 containing copper ions ("pregnant leach  
20 solution") is collected from the bottom of the ore heap or dump 14 and passed through a copper recovery plant 22, where



70-90% of the copper ions are removed, depending on the efficiency of the plant. After exiting the copper recovery plant 22, a copper depleted lixiviant 26 ("raffinate") is collected in containment ponds or sent, via a pump station,  
5 directly back to the top of the ore dumps for additional leaching of copper. Sulfuric acid addition is sometimes required to the raffinate.

In the process, the "raffinate" 26 is processed through a microfiltration or ultrafiltration membrane system 30.  
10 The membrane system separates the raffinate into two streams: permeate 34 and concentrate 38. The concentrate 38 consists of substantially all of the entrained organic collector in the raffinate. The permeate 34 consists of a substantially organic - free solution to be sent directly to  
15 the ore heap for leaching.

The concentrate 38 may be sent to a raffinate collection pond, used to leach specific ore heaps or dumps, or be removed from the leaching circuit. It may also be further processed using a phase separation tank to skim off  
20 the concentrated organic.

Presently, copper mining operations are discharging

raffinate with little or no attempt to recover entrained organic. Economic losses of organic have been reported as \$1-2 million dollars/year for medium sized copper SX-EW facilities. This demonstrates that using a membrane system  
5 to recover the entrained organic offers significant, direct process and operating cost advantages. In addition, the elimination of organic coating of the ore to be leached has a positive effect on leaching efficiency.

The membrane system in question would process 100 -  
10 20,000 gallons per minute of raffinate, with 80-95% of the feed flow becoming permeate product. Typical microfiltration and ultrafiltration membranes used would be G, J, K, and DS-7 series elements from Osmonics/Desalination Systems of Vista, CA. These spiral wound elements use poly  
15 acrylonitrile, PTFE (Teflon), PVDF, and/or polyarimid membrane materials. The described membranes span the microfiltration/ultrafiltration membrane category, with molecular weight cut-offs of 5,000 to 100,000 MWCO and pore sizes of 0.003 microns to 0.1 micron.

20 A typical system would process 1,000 gpm of raffinate through 348 each 8 inch spiral wound JX membrane elements.

The system would split the feed flow into 900 gpm of permeate and 100 gpm of concentrate. The concentrate would be removed from the leaching circuit, or processed through a phase separation system to recover the organic and the concentrate returned to the leaching system. The permeate, containing no organic, would be returned to the top of the ore heap or dump for enhanced leaching of copper.

What is claimed is:

1. A process for recovering a metal from a metal-containing material, comprising:

(a) contacting a metal leaching agent with the metal-containing material to solubilize the metal in a pregnant

5 leach solution;

(b) contacting the pregnant leach solution with an organic collector to transfer at least a portion of the solubilized metal to the organic collector to form a pregnant organic solution including a metal-containing  
10 organic collector and a stripped pregnant leach solution, wherein the stripped pregnant leach solution contains at least a portion of the organic collector;

(c) recovering the metal from the metal-containing organic collector; and

15 (d) filtering the stripped pregnant leach solution with a filter to form a concentrate containing at least most of the organic collector and a permeate.

2. The process of Claim 1, further comprising:

(a) contacting the permeate with metal-containing

material.

3. The process of Claim 1, further comprising:

(a) recovering the organic collector from the concentrate.

4. The process of Claim 1, wherein the metal is selected from the group consisting of copper, nickel, cobalt, zinc and uranium, and mixtures thereof.

5. The process of Claim 1, wherein the organic collector is selected from the group consisting of LIX 63, LIX 64, LIX 65, LIX 85 and LIX 860 as manufactured by HORNKREL of Tucson, Arizona, PT5050, M5640 and M56115 as

5 manufactured by ACORGA, LTD., and mixtures thereof.

6. The process of Claim 1, wherein the leaching agent is selected from the group consisting of sulfuric acid, a chloride, a nitrate, ammonia, ammonium salts, a sulfate, and mixtures thereof.

7. The process of Claim 1, wherein the filter has a pore size ranging from about 0.003 to about 0.1 microns.

8. The process of Claim 1, wherein the concentrate constitutes no more than about 20% of the stripped pregnant leach solution.

9. The process of Claim 1, wherein the permeate constitutes at least about 80% of the stripped pregnant leach solution.

10. The process of Claim 1, wherein the permeate comprises at least most of the leaching agent in the stripped pregnant leach solution.

11. The process of Claim 1, wherein the permeate comprises at least about 80% of the leaching agent in the stripped pregnant leach solution.

12. The process of Claim 1, wherein the concentrate comprises at least about 90% of the organic collector in the

stripped pregnant leach solution.

13. A process for recovering a metal from a metal-containing material, comprising:

(a) contacting a pregnant metal-containing solution with an organic collector to transfer at least a portion of a solubilized metal in the pregnant metal-containing solution to the organic collector to form a pregnant organic solution including a metal-containing organic collector and a stripped solution, wherein the stripped solution contains at least a portion of the organic collector;

(c) recovering the metal from the metal-containing organic collector; and

(d) filtering the stripped solution with at least one of a microfilter and nanofilter to form a concentrate containing at least most of the organic collector and a permeate.

## ABSTRACT

A treatment system for removing an organic material (e.g., an organic lixiviant or collector) from a copper leaching solution. A copper leaching aqueous stream  
5 containing entrained organic material, i.e. "raffinate" from a copper extraction process, is passed through a microfiltration or ultrafiltration membrane system to produce an organic rich concentrate and an organic free permeate. The permeate is returned to the copper ore dump  
10 or heap for enhanced leaching of copper ions from the copper ore. The concentrate may be removed from the leaching circuit or the entrained organic portion of the concentrate may be recovered and recycled back to the head of the solvent extraction process. The result of this membrane  
15 filtration of the copper leaching raffinate is recovery of organic material previously lost to raffinate "carry-over", which can be a large, direct economic benefit. In addition, this process eliminates a potential environmental problem of organic deposition on the ore heap, and avoids coating of  
20 the ore to be leached with an organic material, preventing effective leaching.



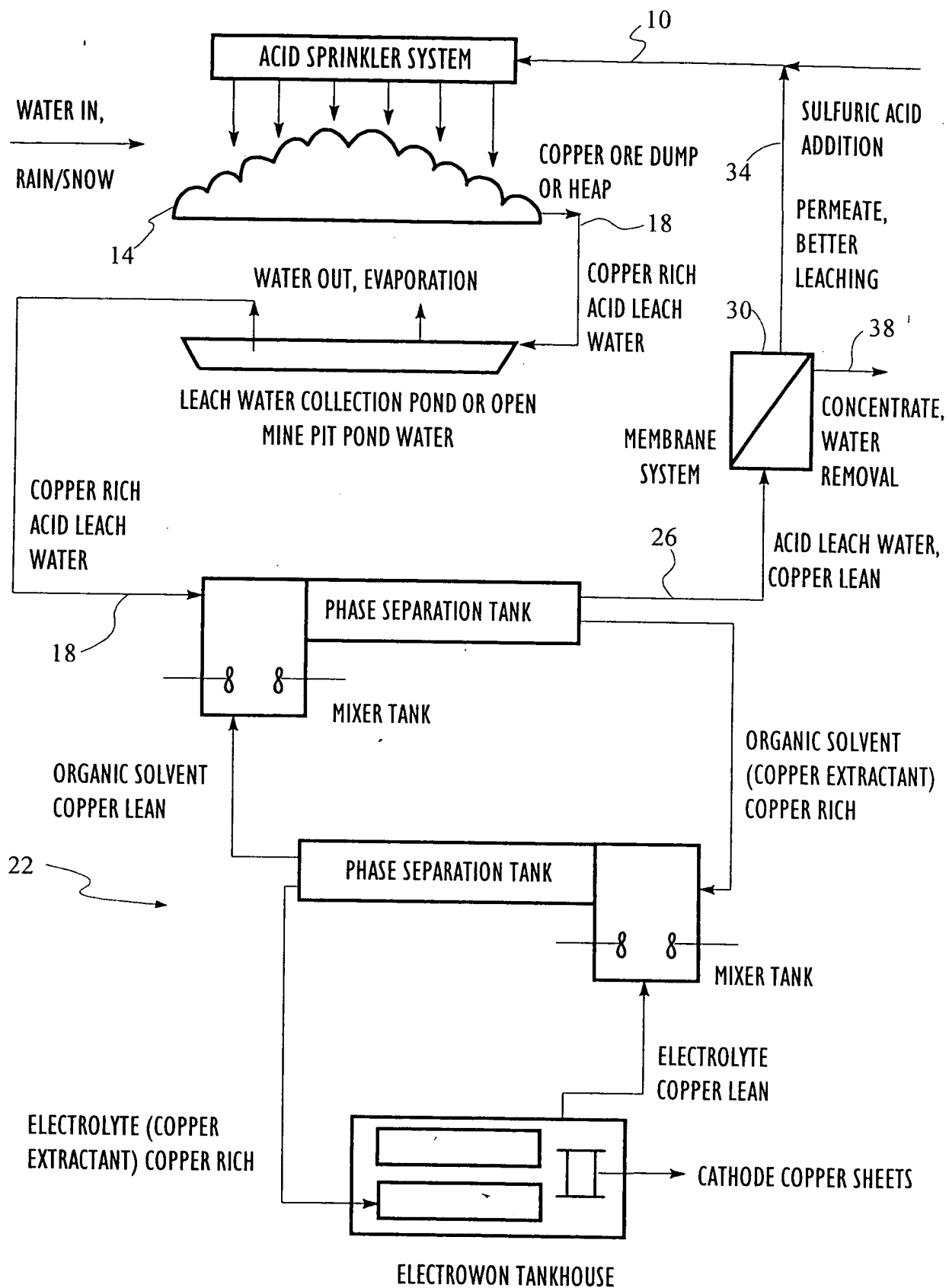


FIG. 1

**VERIFIED STATEMENT (DECLARATION) CLAIMING SMALL ENTITY STATUS  
(37 CFR 1.9(f) and 1.27(c)) - SMALL BUSINESS CONCERN**

I hereby declare that I am an official empowered to act on behalf of the small business concern, **HW PROCESS TECHNOLOGIES, INC.** of 1201 Quail Street, Lakewood, Colorado 80215.

I hereby declare that the above-identified small business concern qualifies as a small business concern as defined in 13 CFR 121.3-18, and reproduced in 37 CFR 1.9(d), for purposes of paying reduced fees under section 41(a) and (b) of Title 35, United States Code, in that the number of employees of the concern, including those of its affiliates, does not exceed 500 persons. For purposes of this statement, (1) the number of employees of the business concern is the average over the previous fiscal year of the concern of the persons employed on a full-time, part-time or temporary basis during each of the pay periods of the fiscal year, and (2) concerns are affiliates of each other when either, directly or indirectly, one concern controls or has the power to control the other, or a third party or parties controls or has the power to control both.

I hereby declare that rights under contract or law have been conveyed to and remain with the small business concern identified above with regard to the invention, entitled "METHOD OF REMOVING ORGANIC LIXIVANT FROM COPPER SX-EW RAFFINATE STREAM" and identified as Attorney File No. 3376-18PROV, described in application Serial No. to be assigned, filed concurrently herewith.

If the rights held by the above-identified small business concern are not exclusive, each individual, concern or organization having rights to the invention is listed below\* and no rights to the invention are held by any person, other than the inventor, who could not qualify as a small business concern under 37 CFR 1.9(c) or by any concern which would not qualify as a small business concern under 37 CFR 1.9(d) or a nonprofit organization under 37 CFR 1.9(e).

\*NOTE: Separate verified statements are required from each named person, concern or organization having rights to the invention averring to their status as small entities. (37 CFR 1.27)

NAME \_\_\_\_\_

ADDRESS \_\_\_\_\_

☒ INDIVIDUAL      ☐ SMALL BUSINESS CONCERN      ☐ NONPROFIT ORGANIZATION

I acknowledge the duty to file, in this application or patent, notification of any change in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small entity is no longer appropriate. (37 CFR 1.28(b))

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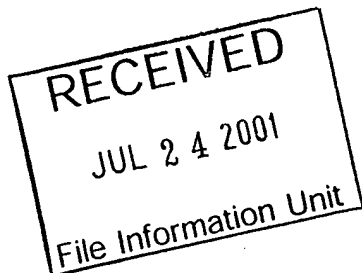
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By: H. E. Wharley

Harold E. Wharley

Vice President Finance and Administration

## REQUEST FOR ACCESS OF ABANDONED APPLICATION UNDER 37 CFR 1.14(a)



In re Application of \_\_\_\_\_

Application Number

Filed

60/064,279

10-30-97

Group Art Unit

Examiner

Paper No. \_\_\_\_\_

Assistant Commissioner for Patents  
Washington, DC 20231

I hereby request access under 37 CFR 1.14(a)(3)(iv) to the application file record of the above-identified ABANDONED application, which is: (CHECK ONE)

☒ (A) referred to in United States Patent Number 6,561,861 column \_\_\_\_\_☐ (B) referred to in an application that is open to public inspection as set forth in 37 CFR 1.11, i.e., Application No. \_\_\_\_\_ filed \_\_\_\_\_ on page \_\_\_\_\_ of paper number \_\_\_\_\_☐ (C) an application that claims the benefit of the filing date of an application that is open to public inspection, i.e., Application No. \_\_\_\_\_ filed \_\_\_\_\_ or☐ (D) an application in which the applicant has filed an authorization to lay open the complete application to the public.

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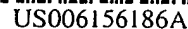
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**(11) Patent Number: 6,156,186**

[45] Date of Patent: \*Dec. 5, 2000

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- [57]
- ABSTRACT**

The present invention is directed to a process for removing various contaminants (e.g., organic collectors, contaminant metals or spectator ions, and/or suspended and colloidal solids) from process streams in leaching processes. The contaminant removal is performed by one or more membrane filtration systems (e.g., nanofilters, ultrafilters, and/or microfilters) treating process streams including, the pregnant leaching solution, the barren raffinate, and the lean and rich electrolytes.

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WO 94/27711 12/1994 WIPO.

